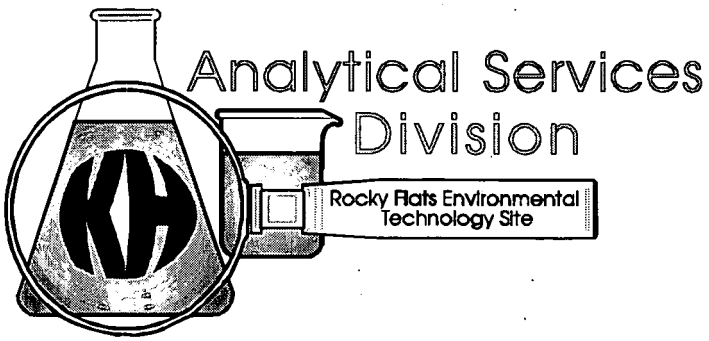


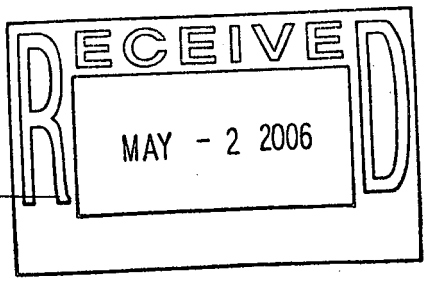
VERIFICATION AND VALIDATION GUIDELINES

FOR

GROSS ALPHA AND GROSS BETA BY GPC

DA-RC04-v2

Effective Date: October 1, 2002



Approved: E A Bowsky
Analytical Services

ADMIN RECORD

Reviewed For Classification
By: Roger S. Cichorz U/NU
ASD Project Lead – T130C
Date: June 4, 2002

1/22

TABLE OF CONTENTS

V & V GUIDELINE CHANGE DESCRIPTION FORM

Instructions: Replace Version 1 with Version 2

Guideline: DA-RC04	**Version:** 2	**Originator:** J.P. Garrett
Description: Verification and Validation. Guidelines for Gross Alpha and Gross Beta by GPC		

Section No.	Change Description
Cover Page	New version and Effective date
Introduction	A new introduction was written to incorporate the BOA SOW rather than PSA Modules.
Entire Document	For clarity, change bars appearing on a Section Title indicate significant changes to the entire Section.
Entire Document	Text reworded for clarity, where the original meaning remains unchanged, will not be identified with change bars.
Entire Document	References to the BOA SOW and the RFETS BOA Implementation document GR03 & GR04, are incorporated throughout the document. References to PSA Modules were eliminated. References to Module Specific Verification and Validation (V & V) Guidelines were replaced with Analytical Specific V & V guidelines.
Data Review Checklist	All references to the Data Review Checklist and its examination were removed from the Guidelines.
Entire Document	Removed Reference to Instrument Calibration Package
Section 2.1, Items 1 & 2	Changed wording to reflect requirement of acidification of aqueous samples only
Section 2.1, Item 3	Included a check for compliance to the 180 holding time.
Section 2.2	The section for reviewing the Sample Data Package Narrative requirements was updated to the requirements of the BOA.
Section 2.3, Action 5	Included in action item to reject data if appropriate batch QC samples were not included in re-analysis.
Section 2.4, Action 2a	An action to issue a NCN for missing QC samples was added. Other actions regarding missing QC were clarified.
Section 2.4, Item 6	Count time requirements for QC samples were added to this item.
Section 2.4, Item 6	Added requirement to report the duplicate relative Error Ratio.
Section 2.5, Objective	Expanded the objective for this section to include assessment for sample homogeneity.
Section 2.5, Item 2	A step was added for verification to evaluate the results of the equivalency test.
Section 2.5, Item 3	An calculation of the BOA criterion for the normalized absolute difference between the sample and laboratory duplicate results was added.
Section 2.6, Objective	Expanded the objective for this section to include assessment of the relative bias for method accuracy.
Section 2.6, Item 3	Deleted reference to LCS percent recoveries and added the assessment of the Relative Bias per the requirements of the BOA.
Section 2.6	Deleted reference to OV/SV requirements for LCS

1. INTRODUCTION/SCOPE

This document presents those data assessment steps which are unique to Gross Alpha and Gross Beta by GPC. This Analytical Specific document is to be used in conjunction with DA-GR01, "General Guidelines for data Verification and Validation.

The purpose of this document is to provide guidance in the completion of Data Verification, and Data Validation activities as part of the Rocky Flats Environmental Technology Site (RFETS) Analytical Services Division Data Assessment Process as described in DA-GR01.

This version of DA-RC04 is applicable to Gross Alpha and Gross Beta by GPC Sample Data Packages generated under the National Basic Ordering Agreement (BOA) Statement of Work (SOW) and the Rocky Flats Environmental Technology Site (Site) BOA Implementation Requirements documents, GR03 & GR04.

2. VERIFICATION AND VALIDATION INSTRUCTIONS

The instructions contained in this section are specific to Gross Alpha and Gross Beta determinations by GPC. They are to be used in conjunction with the general instructions for Verification and Validation found in Analytical Services Division's General Guidelines for Verification and Validation, DA-GR01.

2.1. Chain of Custody, Holding Times, and Sample Preservation

Review Items: Sample & QC Result Summary, COC record, and sample preparation raw data.

Objective: To ascertain the validity of results based on the method required sample preservation, and the continuity of sample custody.

Source: GR03 § 6, BOA Attachment 1, § 3.1.2; Attachment J to BOA Attachment 1, § 1.2

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check for documentation that the pH of aqueous samples were adjusted to ≤ 2 prior to receipt by the laboratory.

Action 1: If aqueous samples were not acid-preserved prior to receipt by the laboratory, comment and assign the reason code **[703]** to all applicable samples.

Item 2: Check for documentation showing aqueous samples were adjusted to a pH of ≤ 2 by the laboratory if samples were not adjusted to the proper pH prior to receipt by the laboratory.

Action 2: If an aqueous sample was not adjusted to the proper pH by the laboratory, when appropriate, initiate a Non-Compliance Notification (NCN) and estimated **[J 201]** all applicable results.

Item 3: Verify the maximum hold time of 180 days was not exceeded.

Action 3: If samples were not analyzed within the 180 day hold time, do not qualify the data, comment and assign the reason code **[101]** to all applicable samples.

2.2. Sample Data Package Narrative Requirements

Review Items: Sample Case Narrative

Objective: Review the narrative for compliance to requirements, problems or unusual circumstances encountered in the analytical processing of samples and for information useful to data assessment.

Source: GR03 § 3.2, BOA Attachment 1, § 3.1.6.2

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Check that the SDP Narrative is present and includes the following as applicable:

- Procedures and/or Standard Method reference for preparation and analysis.
- Descriptions of significant technical difficulties encountered in preparing and analyzing the samples.
- Justification of all dilutions.
- Explanations of any QC deficiencies, or inability to achieve the required detection limits (RDLs).
- Reasons for reanalysis, reanalysis Analytical Batch Identifications Numbers, and a synopsis of the reanalysis Analytical Batch QC Assessment.
- Explanations and descriptions of all deviations from routine protocols, including deviations from approved standard operating procedures (SOPs), detection limit modifications, etc. If it was necessary to contact the CTR for instructions due to the nature of the deviation, the laboratory shall document those instructions in the narrative.

Action 1: If any of the above items are non-compliant, do not qualify the results, comment and include the reason codes **[227]** and/or **[805]** as appropriate. Use professional judgement to determine if the issuance of a NCN is warranted.

2.3. Sample & QC Results Summary

Review Items: Sample and QC Results Summary

Objective: Review the Samples section of Samples and QC Sample Results Summary for compliance to requirements and for information useful to data assessment.

Sources: Attachment J to BOA Attachment 1; GR03, § 6

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify all samples and tests that were requested on the COC for gross alpha gross beta determinations have been analyzed, tested and appear on the Sample and QC Result Summary.

Action 1a: If sample were not analyzed, and documentation identifies a valid reason, do not qualify data. Address the deficiency as a comment in the Data Quality Assessment Report.

Action 1b: If the Sample and QC Result Summary is missing or incomplete, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2 Verify the Sample and QC Results Summary contains the following information for Site Samples and laboratory QC samples as applicable:

- LABORATORY NAME
- REPORT IDENTIFICATION NUMBER (RIN)
- RFETS SAMPLE ID
- LAB SAMPLE ID
- ANALYTE
- SAMPLE MATRIX
- RESULTS and UNITS
- 2S(total propagated uncertainty)
- MDA
- ALIQUOT SIZE ANALYZED
- ANALYTICAL BATCH ID

Action 2a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 2b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Verify only one result is reported for each requested analyte.

Action 3: If more than one result is reported and neither is identified as "Do Not Use data", issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received..

Item 4: Verify the MDA for each sample is reported and is ≤ the RDL.

Action 4a: If the MDA is > the RDL, and a reduced aliquot size was used due to high solids or high activity, or insufficient sample volume provided, do not qualify the data.

Acton 4b: If the MDA exceeds the RDL for reasons other than those identified in Action 4a, estimate **[UJ 136]** all applicable data.

Action 4c: If the MDA is > the RDL and the deficiency is not reported in the narrative, comment and assign reason code **[805]** to all applicable data.

Evaluation: ***The following items applies to validation only:***

Item 5: Verify samples requiring reanalysis have been assigned a new analytical batch identification number and appropriate QA/QC is included.

Action 5: If data can not be produced to show the batch ID is different and the appropriate batch QA/QC was included in the reanalysis, reject **[R 205]** all applicable data.

Item 6: If the MDA is > the RDL and the samples and duplicates were prepared with a reduced aliquot size due to high solids or high activity, verify that the following criteria were met:

- The measurement uncertainty is not greater than 10% of the sample activity
- The MDA for the analysis is a maximum of 10% of the sample activity

Note: If the sample was contaminated with both alpha and beta emitting isotopes to approximately the same activity level (order of magnitude), the above criteria are applicable for both the alpha and beta activities. If the sample is primarily contaminated with either alpha or beta emitting isotopes, the above criteria apply to the higher activity analyte.

Action 6: If any of these items are non-compliant, and the MDA > RDL due to laboratory error, use professional judgment to determine the affect of the non compliance on the data. At a minimum, estimate **[UJ 136]** all applicable data.

Item 7: Calculate at least one sample MDA using the following equation:

Note: Except as specified above, the analyte MDAs shall be less than or equal to the respective RDL.

$$MDA = \frac{\frac{2.71}{T_S} + 3.29\sqrt{\frac{BKG}{T_S} + \frac{BKG}{T_B}}}{EFF * SA * AMT * 2.22}$$

where,

BKG = Alpha background count rate (cpm) for alpha MDA. Beta background count rate (cpm) for beta MDA.
TB = Background count time (minutes)
TS = Sample count time (minutes)
EFF = Efficiency of the detector at weight 0. Alpha efficiency for calculation of alpha MDA. Beta efficiency for calculation of beta MDA
AMT = Sample aliquot size (liters or grams)
SA = Self-absorption correction factor at residue weight

Units are in pCi/l or pCi/g

Action 7: If MDAs have been calculated wrong, whether the parameters have been entered wrong or there has been a calculation error, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.4. Batch QC Summary

Review Item: Sample and QC Results Summary, Batch QC Summary, and Raw Data

Objective: Review the QC Sample Results Summary and the Batch QC Summary for compliance to requirements and for information useful to data assessment.

Sources: Attachment J to BOA Attachment 1; GR03, § 6

Evaluation: ***The following items apply to both validation and verification:***

Item 1: Check that the Batch QC Summary is present and complete. The following information shall be included:

- LAB SAMPLE ID
- COUNT DATE
- QC OBSERVED VALUE with associated two sigma uncertainty
- LCS: Know value and relative Bias
- BATCH BLANK: RDL and MDA
- DUPLICATE: For each duplicate pair, the result of the duplicate equivalency test as defined in *BOA Attachment J, Section 2.3.3*

Action 1: If the Batch QC Summary is not present or is missing information required for data assessment, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify a Laboratory Control Sample (LCS), Laboratory Duplicate, and Preparation Blank were included for each analytical batch.

Action 2a: If any of these QC samples are missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables

for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Acton 2b: If any single QC sample is missing and data cannot be obtained from the laboratory, qualify applicable data as follows:

- If missing Duplicate only, estimate **[J 128]** all applicable data.
- If missing LCS only, reject **[R 174]** all applicable data.
- If missing Preparation Blank only, reject **[R 175]** all applicable data.

Action 2c: If more than one QC sample is missing and data cannot be obtained from the laboratory, qualify as rejected **[R 230]** all applicable data.

Item 3: Determine if the QC frequency is met by verifying that each analytical batch contains no more than 20 customer samples per set of QC samples (LCS, Laboratory Duplicate, and Preparation Blank).

Action 3: If this item is non-compliant, qualify as estimated **[J 168]** all applicable data.

Item 4: Verify all QC deficiencies are detailed in the narrative.

Action 4: If this item is non-compliant, do not qualify any data. Comment and assign the reason code **[805]** to all applicable data.

Item 5: Verify all sample results, including reanalysis, and the corresponding Analytical Batch QC sample results were reported.

Action 5a: If this item is non-compliant, address the deficiency in the Data Assessment Report using professional judgment to qualify the data. Omissions or errors which do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 5b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following items apply to validation only:***

Item 6: Verify each QC sample type is clearly identified, i.e., a designator clearly identifies a QC sample as being a LCS, Batch Blank, or Duplicate, and the following criteria are met:

- The QC samples are counted for a sufficient time to meet the required detection limit except in the case where the achieved MDA is calculated from the standard deviation of a blank population in which case the batch blank will be counted for the same count time as the samples.
- For each batch duplicate pair, the following additional information is reported:
  - ◊ result of duplicate result equivalency test as defined in Section 2.5, including calculated values for relative Error Ratio.

- For the "LCS", the following additional information is reported:
  - ◊ LCS "SV" (standard value (SV) of the LCS; decayed to analysis date, if applicable)
  - ◊ Uncertainty of LCS standard value (2-sigma)
  - ◊ LCS Relative Bias

Action 6a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 6b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.5. Duplicate Samples

Review Item: Sample and QC Results Summary, Batch QC Summary, and Raw Data

Objective: To determine a measure of laboratory precision, or degree of agreement of repeated measurements within acceptable concentration ranges and to assess the homogeneity of the samples.

Sources: Attachment J to BOA Attachment 1; GR03, § 6

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify the results for the duplicate are reported separately from the corresponding sample.

Action 1a: If this item is non-compliant, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 1b: If the laboratory is unable to supply the missing data or errors cannot be corrected, do not qualify any data, comment and assign the reason code **[205]** to all applicable data.

Item 2: Verify that the reported duplicate equivalency test is ≤ 3.

Action 2a: If a duplicates exceed the equivalency test requirement of ≤ 3 due to the difficulty of subsampling and this explanation is described in the Case Narrative, no action is taken.

Action 2b: If the duplicate equivalency test does not pass and the sample is homogeneous, estimate **[J 235]** all applicable data in the analytical batch.

Evaluation: ***The following item applies to validation only:***

Item 3: Calculate the normalized absolute difference between the sample and laboratory duplicate results using the following equation and confirm the value reported:

$$\frac{S - D}{\sqrt{(TPU_S)^2 + (TPU_D)^2}} \leq 3 *$$

where:

S	=	Sample result
D	=	Duplicate result
TPU_S	=	1s Total Propagated Uncertainty of the sample
TPU_D	=	1s Total Propagated Uncertainty of the duplicate

(* 2.58 was rounded to 3)

Action 3: If the duplicate equivalency test was calculated wrong, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.6. Laboratory Control Sample Analysis

Review Item: Sample and QC Results Summary, Batch QC Summary, and Raw Data

Objective: To determine the overall performance of each step during sample preparation and analysis, and to evaluate the LCS relative bias as a means of assessing the accuracy of the analytical method.

Sources: Attachment J to BOA Attachment 1; GR03, § 6

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify that the LCS certified activity and uncertainty at the 2-sigma, decayed to analysis date, are reported.

Action 1: If the laboratory control was not reported and/or decay corrected to the analysis date, reject **[R 174/236]** all applicable data.

Item 2: Verify the activity level in the LCS is at an appropriate level for the samples in the Analytical Batch.

The activity in the analysis aliquot should be sufficiently high to produce statistically sound data. However, the activity level should not be so high as to create a potential for sample or laboratory cross-contamination.

- The LCS for low level environmental water samples, low level soil samples, and low level waste samples shall be less than 10 pCi /aliquot.
- For higher level samples, is the activity in the analysis aliquot (not the concentration of the activity in the sample) used to determine the appropriate LCS level

Action 2: If the laboratory control sample does not pass all of these criteria, estimate **[J 234]** all applicable data.

Evaluation: ***The following items applies to validation only:***

Item 3: Verify, by calculation, the LCS relative bias results fall within the range of -.25 to +.25 (Reference: ANSI N13.30, Appendix B) by using the following equation:

$$\textbf{Relative bias} = \frac{observed - known}{known}$$

Action 3: If the laboratory control sample does not pass the relative bias criteria, professional judgment should be used to determine the effect this has on the data. At a minimum, estimate **[J 236]** all applicable data.

Item 4: Verify that the LCS meets the following criteria:

- The isotopes for the LCS are 241Am for gross alpha and 90Sr/90Y or 137Cs for gross beta analyses.
- The units for reporting the LCS are up to the discretion of the laboratory as long as the units are specified, and are the same for both the observed and certified values.
- The LCS shall be counted for the same count duration as the samples
- The LCS was prepared and analyzed in the same manner as the samples.

Action 4: If the laboratory control sample does meet all criteria, estimate **[J 234]** all applicable data.

2.7. Preparation Blank

Review Item: Sample and QC Results Summary, Batch QC Summary, and Raw Data

Objective: To assess the extent of contamination introduced through sample preparation, and analysis.

Sources: Attachment J to BOA Attachment 1; GR03, § 6

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify that the preparation blank meets the following requirements:

- The preparation blank was prepared from ASTM Type II water.
- An aqueous Preparation Blank was used for all aqueous and non-aqueous matrices.
- Preparation blanks were counted for at least the same count duration as the samples unless the samples had to be counted longer than the routine count time in order to meet the RDL.
- The Preparation Blank MDA calculation is based on the greatest sample volume or weight for the entire Analytical Batch.

Action 1: If these items are non-compliant, do not qualify any data. Comment and assign the reason code **[234]**

Evaluation:	***The following items apply to validation only?***
Item 2:	If the MDAs for the samples in an Analytical Batch meet the analyte RDLs and the sample activities are **less than** 5 times the RDL, the activity of the preparation blank shall be equivalent to zero when the measurement uncertainty is considered and shall be less than or equal to the RDL.
Action 2:	If the preparation blank is not equivalent to zero when the measurement uncertainty is considered and is not less than or equal to the RDL, estimate **[J 237]** all applicable data.
Item 3:	If the MDAs for the samples in an Analytical Batch meet the analyte RDLs and the sample activities are **greater than or equal** to 5 times the RDL, the activity of the preparation blank shall be equivalent to zero when the measurement uncertainty is considered.
Action 3:	If the preparation blank is not equivalent to zero when the measurement uncertainty is considered, estimate **[J 237]** all applicable data.
Item 4:	If the MDAs for the samples in an Analytical Batch do not meet the analyte RDLs due to sample matrix effects and the sample activities are less than the MDAs, the Preparation Blank data shall be assessed as follows: • The Preparation Blank analyte activity shall be equivalent to zero when the measurement uncertainty is considered. • The Preparation Blank activities shall be less than the MDAs for the sample measurements.
Action 4:	If the preparation blank is not equivalent to zero when the measurement uncertainty is considered and is not less than the MDA, estimate **[J 237]** all applicable data.
Item 5:	If the MDAs for the samples in an Analytical Batch do not meet the RDL due to high analyte activity and reduced sample size, the Preparation Blank analyte activity shall be less than 1% of the sample analyte activity.
Action 5:	If the Preparation Blank analyte activity is not less than 1% of the sample analyte activity, estimate **[J 237]** all applicable data.
Item 6:	Verify that the activity for the samples and QC samples have **not** been blank corrected.
Action 6:	If the samples and/or the QC samples have been blank corrected, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

2.8. Sample Preparation Raw Data

Review Items: Preparation Raw Data

Objective: To determine if bench sheets and run logs have been filled out properly and if the proper sample preparation methods were performed.

Sources: Attachment J to BOA Attachment 1, GR03, § 6

Evaluation: ***The following items apply to verification and validation:***

Item 1: Verify that benchsheets and/or preparation logs are included in the SDP.

Action 1: If benchsheets and/or preparation logs are not included, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following items apply to validation only:***

Item 2: Verify that benchsheets and/or preparation logs are included and document the required items as follows:

- ANALYTICAL BATCH IDENTIFIER
- DATE OF PREPARATION
- IDENTIFIER FOR THE LABORATORY SOP for the preparation
- IDENTIFIERS FOR ALL SAMPLE AND QC SAMPLES in the batch
- IDENTIFIERS THAT PROVIDE FOR TRACEABILITY of LCS, dilutions used, etc.
- CONCENTRATION OF WORKING STANDARDS used for tracer, LCS, matrix spike, etc.
- VOLUMES OR WEIGHTS OF ADDED LCS ANALYTE(S),. (if the concentration is given in activity per unit weight then the weight added shall be reported; if the concentration is given in activity per unit volume, then the volume added shall be reported)
- SAMPLE ALIQUOT PLATED ON PLANCHET, NET WEIGHT OF RESIDUE ON COUNTING PLANCHET
- BALANCE IDENTIFIERS WITH DATES OF USE(if applicable)
- INITIAL AND FINAL WEIGHTS AND VOLUMES for all samples and QC samples including gross weights, tare weights, and aliquot weights where applicable
- PIPETTE IDENTIFIERS AND DATES OF USE (if applicable)
- COMMENTS describing any significant sample changes or reactions which occur during preparation
- SIGNATURES AND DATES of all analysts and reviewers

Soils, Sediments, Sludges and Solid Waste

- APPROXIMATE SAMPLE VOLUME RECEIVED, THE ALIQUOT SIZE HOMOGENIZED

Action 2a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 2b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: For soils, sediments, sludges and waste (which require homogenizing the sample prior to analysis), verify that the following additional information is reported:

- the approximate sample volume of the gross sample (as received)
- the aliquot size homogenized
- the aliquot size of dried, homogenized sample digested
- the ratio of sample weight as received (wet)/sample weight dried

Action 3: If any of these items are non-compliant, do not qualify any data. Comment and assign the reason code **[240]** to all applicable data.

Item 4: Verify that the volume or weight used to calculate the Preparation Blank activity and MDA (pCi/g or pCi/l) did not exceed the maximum volume or weight of sample for the entire Analytical Batch.

Action 4: If this item is non-compliant, do not qualify any data. Comment and assign the reason code **[234]** to all applicable data.

2.9. Standards Summary Raw Data

Review Items: Standard Summary Raw Data

Objective: To verify that all standards meet the requirements for documentation and traceability.

Sources: Attachment J to BOA Attachment 1, GR03, § 6

Evaluation: ***The following items apply to verification and validation:***

Item 1: Verify that the standard summary raw data is included in the in the SDP.

Action 1: If the standard summary is not included, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following items apply to validation only:***

Item 2: For primary or other standards (both diluted and undiluted) used for LCS and any in-house prepared instrument calibration sources, verify that the following information for the diluted standard preparation is reported as applicable:

- STANDARD ID. (Working Standard) that was used traced back to the *PRIMARY STANDARD ID.* (All identifiers must be traceable to standard reference material certificates. Submit only the first page of the NIST certificate to establish primary standard ID. and/or traceability.
- STANDARD ISOTOPE, CONCENTRATION, AND ERROR IN THE WORKING STANDARD USED
- EXPIRATION DATE of Working Standard
- USE for this standard (tracer, LCS, efficiency, etc.)
- DATE OF PREPARATION
- SUFFICIENT DILUTION data to provide for calculation of the activity

Action 2a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 2b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Items 3: Verify a diluted primary standard and/or a secondary standard calculation.

Action 3: If these calculations do not coincide with the standard values, issue a Non-Compliance Notification and qualify all applicable data **[R 243]**.

Items 4: Verify that all standard identifications are traceable to the primary certificate, which are traceable to NIST.

Action 4: If standards are not traceable to the primary certificate or are not traceable to NIST, issue a Non-Compliance Notification and reject **[R 244]** all applicable data.

Items 5: Verify that all standards and sources traceable to NIST have not expired and are valid.

Action 5: If standards and tracers have expired, reject **[R 219]** all applicable data.

2.10. Calibration Raw Data

Review Items: Calibration Raw Data

Objective: To verify the instrument calibration parameters are within control limits and to establish an analytical curve relating the response of an instrument to a quantifiable characteristic of the analyte in known standards.

Sources: Attachment J to BOA Attachment 1, GR03, § 6

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify the instrument calibration raw data contains raw data for the energy calibration, backgrounds and efficiency determinations.

Action 1: If any part of the instrument calibration raw data is missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 2: Verify the following have been identified in the calibration raw data:

- Identifier for the instrument calibration SOP
- Identification of Software used to produce instrument calibration

Action 2: If any one of the above items are missing, do not qualify the data, issue an NCN to prevent recurrence, comment and assign reason code **[804]** to all data.

Item 3: Verify the Data File Name for the instrument calibration applicable to sample analyzed in this data package is included in the Instrument Calibration Raw Data.

Action 3: If the calibration Data File Name for this calibration is not included, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following items apply to validation only:***

Item 4: Verify the required specified items are included for each gas proportional counter used to report results:

Energy Calibration Data

- Instrument and detector ID(s)
- Date of voltage plateau
- discriminator window settings
- isotopes used

Background Determination

- Date of Background
- Length of background count
- Background counts

Efficiency Determination

- Date of efficiency/self-absorption curves
- Isotopes used
- Graph or equation of self-absorption curve

Daily Check Source & Background Counts

- Instrument Used
- File Name of data collected
- Date of calibration checks
- Output of daily background check and output of daily check source count for each detector used to generate data in the package.
- Current control charts for background and daily check source showing 2 & 3 sigma limits.

Action 4a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 4b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 5: Verify the sources used for the determination of self-absorption and cross talk are of similar isotope content to that of the samples. Valid sources include: Am-241 or Th-230 for alpha and Cs-137 or Sr-90/Y-90 for beta.

Action 5a: If this item is non-compliant, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Action 5b: If the laboratory is unable to supply the missing data or errors cannot be corrected, qualify all applicable data rejected **[R 141]**.

Item 6: Determine if check source is within limits

Action 6a: Use professional judgement to evaluate data associated with check source results that fall between 2 & 3 sigma control limits.

Action 6b: If check source exceeds the 3 sigma control limit, issue a NCN and reject **[R 141]** all applicable data.

2.11. Sample Analysis Raw Data

Review Items: Sample Analysis Raw Data

Objective: To verify sample raw data deliverable requirements have been met and that raw data are present in a form suitable for verification and validation. Verify that the instrument raw data is provided for all reported data and that the data is consistent with the results reported on the summary forms.

Sources: Attachment J to BOA Attachment 1, GR03, § 6

Evaluation: ***The following items apply to both verification and validation:***

Item 1: Verify Sample Analysis raw data is present.

Action 1: If sample analysis raw data is missing, issue a NCN, comment and assign reason code **[801]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Evaluation: ***The following item applies to validation only:***

Item 2: Check that all instrument raw data for the RIN are included and are legible. Verify sample analysis raw data are included for all analyses performed and include the following:

- SAMPLE ID (Site / Laboratory)
- DATE AND TIME of analysis
- COUNT TIME
- DATA FILE NAME
- INSTRUMENT AND DETECTOR ID
- FILE NAME OF BACKGROUND USED
- APPROPRIATE DETECTOR BACKGROUND
- DETECTOR EFFICIENCY FOR THIS SAMPLE
- ANALYTICAL BATCH ID
- SAMPLE ALIQUOT SIZE
- ANALYTE ISOTOPE(S)
- ANALYTE(S) GROSS COUNTS
- BACKGROUND COUNTS (IDENTIFY COUNT TIME OF BACKGROUND)
- ANALYTE(S) NET COUNTS
- CALIBRATION ISOTOPES
- INSTRUMENT RUN LOG for applicable count dates(copy is acceptable)

Action 2a: Omissions or errors that do not have an impact on the assessor's ability to assess the data shall be documented with a comment and assigned the reason code **[804].** An NCN shall be issued to prevent the recurrence of such errors or omissions in future data packages.

Action 2b: For other omissions or errors that impact the assessor's ability to complete the data review, issue a NCN, comment and assign reason code **[803]** to all applicable data. Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 3: Verify there is sufficient raw data included to allow manual calculation of the final sample activity, measurement uncertainty, and MDA. Calculate at least one sample activity and its associated measurement uncertainty and confirm reported values.

Action 3: If there is insufficient raw data or reported values could not be confirmed, issue a NCN, comment and assign reason code **[803]** to all applicable data.

Inspect all other SDP deliverables for missing information and incorporate any deficiencies into the NCN. Discontinue the data assessment until a new data package is received.

Item 4: Verify all QC samples were counted and analyzed in the same manner as the samples in the Analytical Batch, in the same time frame, and using the same instrument calibration parameters, and instrument analysis algorithms.

Action 4: If these items are non-compliant, do not qualify any data. Comment and assign the reason code **[234]** to all applicable data.

3. DATA QUALITY ASSESSMENT REPORT PREPARATION

Prepare a Data Quality Assessment Report according to the General Data Assessment guidelines presented in DA-GR01. A Data Quality Assessment Report template for DV-RC04 is presented as Attachment 1.

4. REFERENCES

- Guidance for Radiochemical Data Validation, Draft RD4, October 4, 1995, prepared by Office of Transportation, Emergency Management & Analytical Services (EM 26), Office of Compliance and Program Coordination, Environmental Management, U.S. Department of Energy.
- Reason Codes for Data Assessment, Analytical Services Document
- RFETS BOA Implementation Requirements, GR03 Version A.5
- RFETS BOA Implementation Requirements, GR04 Version A
- Basic Ordering Agreement (BOA) for Laboratory Analytical Services administered by Westinghouse Savannah River Company on behalf of the Department of Energy.

This page intentionally left blank.

ATTACHMENT 1: DATA QUALITY ASSESSMENT REPORT TEMPLATE

GPC

Data Quality Assessment Report

Rocky Flats Environmental Technology Site

RIN Number	Analytical Method/Analytical Specific Line Item Code		Review Level
Analytical Laboratory	**Assessment Performed by**	**Data Assessment Guideline Identifiers**	**Number of Samples**

Sample Numbers: _____

Quality Control Items	Reviewed (Y or N)	Non-Compliance Identified
General (Cover Page, General SDP, Narrative)		
Chain of Custody, Preservation, and Holdings		
Sample Results		
QC Sample Results		
Duplicate Sample Results		
Laboratory Control Results		
Preparation Blank Results		
Preparation Raw Data		
Standards Summary Raw Data		
Calibration Raw Data		
Sample Analysis Raw Data		
Electronic Data Deliverable EDD		
Structural Requirements		
General Requirements		
Energy Calibration		
Backgrounds		
Efficiency Calibration		
Other:		

Y	Item was reviewed or non-compliance was identified
N	Item was not reviewed or non-compliance was not identified
N/A	Item is not applicable to the Line Item

22/22

GPC
Data Quality Assessment Report
Rocky Flats Environmental Technology Site

Data Assessment results are classified as either Action Items or Comments. Action Items are technical non-compliances that result in qualification of analytical results. Data may be qualified as valid (V), estimated (J), presumptively estimated (NJ), estimated at an elevated level of detection (UJ), or rejected (R). Multiple qualifiers may be associated with any given data point based on the number of problems identified, however, the assigned qualifier is based upon the following hierarchy: R, UJ, NJ, J, V. All data points that are not qualified based upon action items in this report are considered valid (V). Comments are technical non-compliances or contractual non-compliances that do not result in qualification of data.

Action Items:

Comments:

Verification/Validation Signature_____ Date:_____

Reviewer Signature_____ Date:_____
(*Validation Only*)